## Virginia Division of Consolidated Laboratory Services

| SOXHLET EXTRACTION by EPA 3540C REVISION 3 DECEMBER 1996  |                     |                   |   |               |          |
|---|---------------------|-------------------|---|---------------|----------|
| Facility Name:  | VELAP ID            |                   |   |               |          |
| Assessor Name:Analyst Name:   | Inspection Date     |                   |   |               |          |
| Relevant Aspect of Standards  | Method<br>Reference | Y                 | N | N/A           | Comments |
| Records Examined: SOP Number/ Revision/ Date  |                     |                   |   | —<br>∖nalyst: |          |
| Sample ID: Date of Sample Preparation   | n:                  | Date of Analysis: |   |               |          |
| Was sodium sulfate (Na <sub>2</sub> SO <sub>4</sub> ) either heated at 400°C for 4 hours or precleaned with methylene chloride prior to use?  | 5.3                 |                   |   |               |          |
| If Na <sub>2</sub> SO <sub>4</sub> was precleaned with methylene chloride, did method blanks demonstrate that Na <sub>2</sub> SO <sub>4</sub> was free from interferences?  | 5.3                 |                   |   |               |          |
| Were sediment/soil samples mixed thoroughly?  | 7.1.1               |                   |   |               |          |
| Were only solids subjected to this extraction procedure?  | 7.1.2               |                   |   |               |          |
| Were dry wastes amenable to grinding ground or sieved so that they could be passed through 1-mm holes?  | 7.1.3               |                   |   |               |          |
| Were gummy, fibrous, or oily materials not amenable to grinding cut, shredded, or otherwise reduced in size to allow for mixing and maximum exposure of sample surfaces during extraction?                          | 7.1.4               |                   |   |               |          |
| When analytes were determined on a dry weight basis, were second portions of samples subjected to drying overnight at 105°C to determine the % dry weight? (The oven-dried portion may not be used for extraction.) | 7.2                 |                   |   |               |          |
| Were weighed portions of sample blended with weighed portions of anhydrous sodium sulfate?  | 7.3                 |                   |   |               |          |
| Were surrogate standard solutions and any matrix spiking solution added onto the samples at this point?   | 7.3.1<br>7.3.2      |                   |   |               |          |
| Were sample sodium sulfate blends extracted with extraction solvent for 16 – 24 hours at 4-6 cycles per hour?   | 7.4                 |                   |   |               |          |
| Were soil/sediment and aqueous sludges extracted with either 1:1 Acetone/Hexane or 1:1 methylene chloride/acetone?  | 5.4.1               |                   |   |               |          |
| Were other samples extracted with either methylene chloride or 10:1 toluene/methanol?   | 5.4.2               |                   |   |               |          |
| Notes/Comments:   |                     |                   |   |               |          |
|   |                     |                   |   |               |          |

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| Sample ID: Date of Sample Preparatio   | n:                  | _ D | ate | of Anal       | lysis:   |
| Were extracts dried by passing through drying columns containing anhydrous sodium sulfate?                                   | 7.8                 |     |     |               |          |
| Were extracts next concentrated?   | 7.9                 |     |     |               |          |
| If necessary, were solvent exchanges as indicated by Table 1 of the reference method followed by another concentration step? | 7.10                |     |     |               |          |
| Were any reagent blanks, matrix spikes, and duplicate samples subjected to all the same procedures as samples?               | 8.1                 |     |     |               |          |
| Notes/Comments:  |                     |     |     |               |          |

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TABLE 1
SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

| eterminative<br>method | Extractio <sup>နိက်</sup> /<br>pH | Exchange<br>solvent<br>for analysis | Exchange<br>solvent<br>for cleanup | Volume of<br>extract for<br>cleanup (mL) | Final<br>extract<br>volume for<br>analysis (mL) <sup>a</sup> |
|------------------------|-----------------------------------|-------------------------------------|------------------------------------|--|--|
| 8041                   | as received                       | 2-propanol                          | hexane                             | 1.0                                      | 1.0, 0.5 <sup>b</sup>  |
| 8061                   | as received                       | hexane                              | hexane                             | 2.0                                      | 10.0   |
| 8070                   | as received                       | methanol                            | methylene chloride                 | 2.0                                      | 10.0   |
| 8081                   | as received                       | hexane                              | hexane                             | 10.0                                     | 10.0   |
| 8082                   | as received                       | hexane                              | hexane                             | 10.0                                     | 10.0   |
| 8091                   | as received                       | hexane                              | hexane                             | 2.0                                      | 1.0  |
| 8100                   | as received                       | none                                | cyclohexane                        | 2.0                                      | 1.0  |
| 8111                   | as received                       | hexane                              | hexane                             | 2.0                                      | 10.0   |
| 8121                   | as received                       | hexane                              | hexane                             | 2.0                                      | 1.0  |
| 8141                   | as received                       | hexane                              | hexane                             | 10.0                                     | 10.0   |
| 8270°                  | as received                       | none                                | -                                  | -  | 1.0  |
| 8310                   | as received                       | acetonitrile                        | -                                  | -  | 1.0  |
| 8321                   | as received                       | methanol                            | -                                  | -  | 1.0  |
| 8325                   | as received                       | methanol                            | -                                  | -  | 1.0  |
| 8410                   | as received                       | methylene chloride                  | methylene chloride                 | 10.0                                     | 0.0 (dry)  |

For methods where the suggested final extract volume is 10.0 mL, the volume may be reduced to as low as 1.0 mL to achieve lower detection limits.

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Phenols may be analyzed by Method 8041, using a 1.0-mL 2-propanol extract by GC/FID. Method 8041 also contains an optional derivatization procedure for phenols which results in a 0.5-mL hexane extract to be analyzed by GC/ECD.

The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

## METHOD 3540C SOXHLET EXTRACTION

